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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) <p>The synthesis of $KAl(CH_2SiMe_3)_2$ by a reductive elimination reaction between $Al(CH_2SiMe_3)_3$ and KH has been attempted but a pure product could not be isolated. The reactants combine at 70°C to form $KAl(CH_2SiMe_3)_3H$, a white pyrophoric solid, which has been fully characterized. A molecular weight</p>		

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study indicates the presence of a mixture of monomeric and dimeric species in benzene solution. The pyrolysis of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ at 205°C produces $\text{Si}(\text{CH}_3)_4$ but also CH_4 . The formation of $\text{Si}(\text{CH}_3)_4$ suggests that $\text{KAl}(\text{CH}_2\text{SiMe}_3)_2$ might have been formed but the presence of CH_4 indicates that a decomposition reaction occurs either prior to or after the formation of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_2$. The reactions of mixtures of $\text{KH-Al}(\text{CH}_3)_3$, $\text{KH-Ga}(\text{CH}_3)_3$, $\text{NaH-Ga}(\text{CH}_3)_3$, $\text{KH-In}(\text{CH}_3)_3$ and $\text{NaH-In}(\text{CH}_3)_3$ have also been investigated in order to determine the role of the organic substituent in the attempted reductive elimination reactions. In all cases the formation of CH_4 during pyrolysis of $\text{KAl}(\text{CH}_3)_3\text{H}$, $\text{KGa}(\text{CH}_3)_3\text{H}$ and the other reaction mixtures is consistent with the occurrence of reduction but no pure compounds could be isolated. A new synthetic route to $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ from AlBr_3 and $\text{LiCH}_2\text{SiMe}_3$ in refluxing hexane is also described.

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TECHNICAL REPORT NO. 8

The Attempted Syntheses of Low Oxidation State, Organometallic Derivatives
of Aluminum, Gallium and Indium, A New Synthesis of $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$

by

O. T. Beachley, Jr., C. Tessier-Youngs, R. G. Simmons and R. B. Hallock

Prepared for Publication

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Buffalo, New York 14214

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[Contribution from the Department of Chemistry,
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The Attempted Syntheses of Low Oxidation State, Organometallic Derivatives
of Aluminum, Gallium and Indium, A New Synthesis of $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$

by

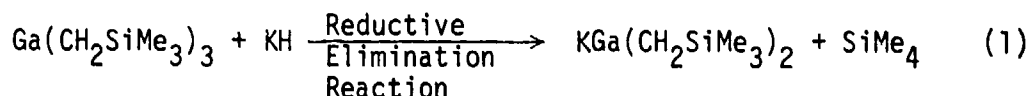
O. T. Beachley, Jr., C. Tessier-Youngs, R. G. Simmons and R. B. Hallock

Abstract

The synthesis of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_2$ by a reductive elimination reaction between $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ and KH has been attempted but a pure product could not be isolated. The reactants combine at 70°C to form $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$, a white pyrophoric solid, which has been fully characterized. A molecular weight study indicates the presence of a mixture of monomeric and dimeric species in benzene solution. The pyrolysis of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ at 205°C produces $\text{Si}(\text{CH}_3)_4$ but also CH_4 . The formation of $\text{Si}(\text{CH}_3)_4$ suggests that $\text{KAl}(\text{CH}_2\text{SiMe}_3)_2$ might have been formed but the presence of CH_4 indicates that a decomposition reaction occurs either prior to or after the formation of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_2$. The reactions of mixtures of $-\text{Al}(\text{CH}_3)_3$, $\text{KH}-\text{Ga}(\text{CH}_3)_3$, $\text{NaH}-\text{Ga}(\text{CH}_3)_3$, $\text{KH}-\text{In}(\text{CH}_3)_3$ and $\text{NaH}-\text{In}(\text{CH}_3)_3$ have also been investigated in order to determine the role of the organic substituent in the attempted reductive elimination reactions. In all cases the formation of CH_4 during pyrolysis of $\text{KAl}(\text{CH}_3)_3\text{H}$, $\text{KGa}(\text{CH}_3)_3\text{H}$ and the other reaction mixtures is consistent with the occurrence of reduction but no pure compounds could be isolated. A new synthetic route to $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ from AlBr_3 and $\text{LiCH}_2\text{SiMe}_3$ in refluxing hexane is also described.

Introduction

An interesting new area of research activity centers on the chemistry of the organometallic derivatives of main-group metals in their low oxidation states. In Group 3 chemistry the list of low oxidation state compounds which are thermally stable at room temperature is very limited but includes $\text{KGa}(\text{CH}_2\text{SiMe}_3)_2$,¹ $\text{NaIn}(\text{CH}_2\text{SiMe}_3)_2$,² $\text{In}(\text{C}_5\text{H}_5)$,^{3,4} $\text{In}(\text{C}_5\text{H}_4\text{CH}_3)$ ⁴ and $\text{Tl}(\text{C}_5\text{H}_5)$.⁵ The cyclopentadienyl-indium and -thallium compounds have been known for many years. In contrast the gallium(I) and indium(I) trimethylsilylmethyl derivatives are new compounds which are prepared by a novel reduction reaction (eq. 1). All data^{1,2} are consistent with the



hypothesis that the preparative reaction involves a reductive elimination reaction of a hydride intermediate, $\text{K}[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3\text{H}]$ or $\text{Na}[\text{In}(\text{CH}_2\text{SiMe}_3)_3\text{H}]$. The synthetic procedures for the reactions are experimentally attractive, require mild conditions (30-50°C in benzene) and lead to the quantitative formation of products.

One goal of our research program is to determine whether the apparent reductive elimination reaction is a general route to low oxidation state, main-group organometallic compounds. The potential variables in the reaction scheme are the central main-group atom, the alkali metal hydride and the organic substituent. The initial investigation of the reduction of $\text{In}(\text{CH}_2\text{SiMe}_3)_3$ with an alkali metal hydride indicated that

the desired reaction occurred very readily.¹ Similarly, the gallium(III) derivative² was converted to the gallium(I) anion without any difficulty or unusual conditions. Our research had also indicated that the specific alkali metal hydride influenced the ease of the reaction. Of the hydrides studied, KH was more reactive than NaH or LiH. The obvious direction of our research was to extend the reduction reaction to aluminum chemistry. Consequently, we have studied the reaction of $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ with KH to determine whether $\text{KAl}(\text{CH}_2\text{SiMe}_3)_2$ could be synthesized. We also wanted to determine whether the organic substituent played an active role in determining the relative ease of the reductive elimination reaction. Therefore, we investigated the potential of reaction mixtures of $\text{In}(\text{CH}_3)_3$, $\text{Ga}(\text{CH}_3)_3$ and $\text{Al}(\text{CH}_3)_3$ with alkali-metal hydrides to undergo the reductive elimination reaction.

Experimental Section

All compounds described in the investigation were extremely oxygen and moisture sensitive and were manipulated in a vacuum line or a purified argon atmosphere. The solvents were purified by refluxing with an appropriate drying agent and vacuum distilled just prior to use. Lithium metal and the alkali-metal hydrides were purchased from Alfa Inorganics as oil dispersions. The oil was removed by repeated extractions with dry pentane or hexane. (Trimethylsilyl)methyl lithium,⁶ $\text{LiCH}_2\text{SiMe}_3$, was prepared from $\text{Me}_3\text{SiCH}_2\text{Cl}$ and Li in either diethylether or hexane and purified by sublimation at 90°C. Trimethylgallium, $\text{Ga}(\text{CH}_3)_3$, was purchased from Alfa Inorganics. Trimethylindium, $\text{In}(\text{CH}_3)_3$, was prepared from InI_3

and CH_3MgI in diethylether and purified.⁷ Methane and hydrogen formed in pyrolysis reactions were measured with a Toepler pump and gas burette assembly.

Characterization Methods. New compounds were analyzed for their Group 3 metal content, bound organic substituents and hydrogen. The Group 3 metals were determined by EDTA titration.⁸ The bound organic ligand and the hydride ligand were converted to the corresponding alkane or H_2 by acid hydrolysis, purified and quantitatively measured by mass or volume measurements. Molecular weight measurements were obtained cryoscopically in benzene by using an instrument similar to that described by Shriver.⁹ The infrared spectra were recorded in the range $4000\text{--}250\text{ cm}^{-1}$ by means of a Perkin-Elmer Model 457 spectrometer. The spectra were recorded as Nujol mulls by using CsI plates. Bands due to mulling agents have been omitted. The ^1H NMR spectra were recorded at 90 MHz and ambient temperature by using a Varian EM-390 spectrometer. All chemical shifts (τ) are given in ppm and are referenced to tetramethylsilane as 10.00 ppm.

Synthesis of $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$. - Tris(trimethylsilyl)methylaluminum, $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$, was prepared from AlBr_3 and $\text{LiCH}_2\text{SiMe}_3$ in hexane. In the drybox, 6.74g (71.7 mmol) $\text{LiCH}_2\text{SiMe}_3$ and 70 mL hexane were combined in an addition funnel equipped with a high vacuum Teflon valve and a stirring bar. Hexane (100 mL) was distilled into a 3-neck 250 mL flask equipped with a stirring bar and charged with 6.39g (23.9 mmol) AlBr_3 . A reflux condenser and the addition funnel were placed on the flask

under a flow of argon. The stirring bar in the dropping funnel was agitated to insure complete dissolution of $\text{LiCH}_2\text{SiMe}_3$ in hexane, and then this solution was slowly added to the stirred AlBr_3 suspension. An exothermic reaction immediately took place precipitating LiBr . The reaction mixture was refluxed for 12 hr. and then the solvent was removed by vacuum distillation. In the dry box the reaction flask was connected to a 2-necked flask by a "90° tube". Using an oil bath at 70°C, 5.885g (20.4 mmol) $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ (85.4% yield) was distilled from the reaction vessel under high vacuum and collected in the 2-necked flask. $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$: BP: 48-49°C/0.01 mm (lit⁶ 51°C/0.08 mm); ^1H NMR (CH_2Cl_2), τ , ppm: 9.93, CH_3 ; 10.24, CH_2 ; IR (Nujol solution, cm^{-1}): 1265 (m, sh), 1253 (vs), 958 (s), 925 (s), 860 (vs), 832 (vs), 764 (vs), 736 (s), 693 (m), 662 (m), 590 (m).

Synthesis of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$. The compound, $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$, was prepared by the reaction of $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ and KH . The reagents, $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ (1.08g, 3.74 mmol) and 0.17g (4.24 mmol) KH were combined in a break-seal tube equipped with a stirrer. Toluene (10 ml) was added by vacuum distillation; the tube was sealed at -196°C and then heated at 70°C for 24 hr. The extent of reaction was monitored by observing the amount of insoluble KH . At the end of the reaction most of the solid KH had disappeared. The reaction tube was opened; the reaction mixture was filtered by means of a medium frit; the solvent was removed by vacuum distillation and 1.11g (3.37 mmol, 90.2% yield) of crude $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ (mp. 138-141°C) was isolated as a white solid. Traces of unreacted

$\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ were removed by washing the product with a small quantity of hexane at -20°C . $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$: MP: $144-146^\circ$, decomposes at 205°C . Anal. Calc: Al, 8.12; H, 1.00 mol/mol; SiMe_4 , 3 mol/mol. Found: Al, 8.19; H, 0.99 mol/mol; SiMe_4 , 2.94 mol/mol. ^1H NMR (benzene, τ (ppm)): 9.59 (s) CH_3 , 10.98 (s) CH_2 . IR (Nujol mull, cm^{-1}): 1560 (w, br), 1250 (s), 1161 (w), 1024 (m), 944 (s), 920 (s), 861 (vs) 839 (vs), 761 (s), 736 (s), 684 (m), 564 (m). Cryoscopic molecular weight. Formula weight: 328.7. Molality, obs. mol. wt., association: 0.0874, 585, 1.78; 0.0556, 526, 1.60; 0.0436, 503, 1.53; 0.0290, 421, 1.28. Solubility: soluble in ethers and aromatic hydrocarbons; moderately soluble in aliphatic hydrocarbons.

Synthesis of $\text{KGa}(\text{CH}_3)_3\text{H}$. The compound, $\text{KGa}(\text{CH}_3)_3\text{H}$, was prepared by the reaction of $\text{Ga}(\text{CH}_3)_3$ and KH in dimethoxyethane or benzene. The reagents, $\text{Ga}(\text{CH}_3)_3$ (1.000g, 8.74 mmol) and KH (1.051g, 26.22 mmol) were combined in a break-seal tube. Dimethoxyethane (25 ml) was added by vacuum distillation. After 96 hr. at room temperature, the reaction mixture was filtered and the solvent was removed to leave $\text{KGa}(\text{CH}_3)_3\text{H}$ (1.250g, 8.08 mmol), 92.5% yield). $\text{KGa}(\text{CH}_3)_3\text{H}$: MP: $150-152^\circ\text{C}$. Anal. Calc. Ga 45.04. Found 45.16. ^1H NMR (dimethoxyethane, τ (ppm)): 10.87, 10.93. (Width at half-heights are 4 and 6 Hz, respectively.) IR (Nujol mull): 2292 (m), 1254 (m), 1200 (m), 1161 (s), 1085 (s), 1030 (m), 990 (w), 847 (vs), 827 (vs), 700 (s), 577 (m), 555 (m), 512 (vs), 482 (vs).

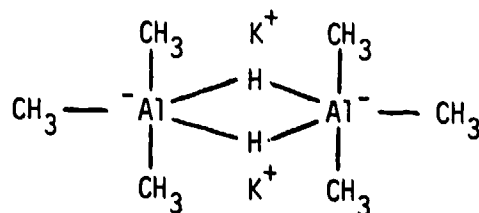
Results and Discussion

The organoaluminum compound $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$, the starting reagent for our attempted synthesis of an aluminum(I) species, has been previously reported.¹⁰ However, it was prepared by a tedious reaction procedure. A 14 day reflux period for a mixture of $\text{Hg}(\text{CH}_2\text{SiMe}_3)_2$, Al foil and toluene was required. Consequently, a more convenient route to $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ which avoided the long reaction time and the toxicity of the organo-mercury compound was developed. The two other common routes to organo-Group 3 compounds use Grignard reagents and lithium alkyls. The analogous gallium¹ and indium² compounds have been synthesized by the reaction of the Grignard reagent, $\text{Me}_3\text{SiCH}_2\text{MgCl}$, with GaCl_3 or InI_3 in diethyl ether. The reaction of the Grignard reagent with AlCl_3 produces the etherate, $(\text{Me}_3\text{SiCH}_2)_3\text{AlO}(\text{C}_2\text{H}_5)_2$, a consequence of the high Lewis acidity of aluminum.¹¹ Therefore, a high yield synthesis of $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ from the $\text{LiCH}_2\text{SiMe}_3$ ⁶ and AlBr_3 in hexane was developed. When the reaction mixture was refluxed for 12 hr, $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ was isolated by vacuum distillation in 85% yield. If the reaction mixture was not refluxed, the organoaluminum product contained halogen atoms. It is of interest to note that the bulky trimethylsilylmethyl substituent leads to increased hydrolytic stability of $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ when compared to $\text{Al}(\text{CH}_3)_3$ and $\text{Al}(\text{C}_2\text{H}_5)_3$.

The synthesis of an aluminum compound in a formal low oxidation state has been attempted but without complete success. No pure, well-characterized organoaluminum(I) compound could be isolated. The research

plan was to combine $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ with KH in a hydrocarbon solvent and to elevate the temperature. The formation of $\text{Si}(\text{CH}_3)_4$ would be consistent with the occurrence of a reductive elimination reaction analogous to that observed in gallium¹ and indium² chemistry (eq. 1). We had every reason to believe that the desired reductive-elimination reaction would occur. The literature even had an example of a reductive elimination reaction in aluminum chemistry.¹² The thermal decomposition of LiAlH_4 at 187-218°C has been reported to produce LiAlH_2 and H_2 . However, our experimental data suggest that $\text{KAl}(\text{CH}_2\text{SiMe}_3)_2$ is not the final product because extensive decomposition involving the trimethylsilylmethyl substituent also occurs.

The reaction of $\text{Al}(\text{CH}_2\text{SiMe}_3)_3$ and KH in toluene at 70°C gave $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$, the apparent intermediate for the reductive elimination reaction, in very high yield. This presumably "ionic" compound exists at room temperature as a colorless, pyrophoric solid and has considerable solubility in hydrocarbon solvents. Cryoscopic molecular weight studies in benzene suggest that $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ exists in solution as a mixture of monomeric and dimeric species. The apparent association in benzene varies from 1.28 to 1.78 in the concentration range of 0.029-0.087 m. A dimeric structure which involves hydride bridges and five coordinate



aluminum would be consistent with the observed association. The infrared spectrum of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ as a Nujol mull supports the proposed structure. There is a broad band at 1560 cm^{-1} which is assigned to the aluminum-hydrogen stretching motion. The ^1H NMR spectrum is not helpful in learning more about the solution structure of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ but it is reported in the Experimental section.

When $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ was heated, melting occurred at $144\text{--}146^\circ$ but decomposition does not begin until 205°C as shown by bubbling and formation of an off-white solid. The stoichiometry of the reaction reveals that one mole of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ forms $1.53\text{ mol Si}(\text{CH}_3)_4$, 0.50 mol CH_4 and an air-sensitive, fluffy white solid, which is insoluble in all non-reactive solvents including hydrocarbons and ethers. The properties of the solid prevented further identification. No data could be used to confirm the formation of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_2$. The pyrolysis product has no infrared bands characteristic of an Al-H bond. The formation of $1\text{ mol Si}(\text{CH}_3)_4$ would be consistent with the proposed reductive elimination reaction (eq. 1). However, formation of CH_4 and the extra $0.53\text{ mol Si}(\text{CH}_3)_4$ per mol $\text{KAl}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ suggest that the ligand has been involved in some type of decomposition reaction. There are no data which can be used to determine whether the proposed decomposition occurs prior to or after the formation of $\text{KAl}(\text{CH}_2\text{SiMe}_3)_2$.

The observed decomposition of the trimethylsilylmethyl groups and our goal to determine the role of the organic substituent in the reductive elimination reaction prompted us to study the thermal reactions of $\text{KAl}(\text{CH}_3)_3\text{H}$. This compound was prepared using the method of Zakharkin

and Gavrilenko.¹³ The colorless compound does not melt but begins to decompose at 200°C. After 4-5 hr. at this temperature, gas evolution appears to stop and 0.933 mol CH₄/mol KAl(CH₃)₃H is formed. The product, a highly air-sensitive grey solid, is insoluble in hydrocarbon and ether solvents. Consequently, further identification of the material was difficult. The nearly stoichiometric evolution of methane could be consistent with the formation of an aluminum(I) compound by the reductive elimination reaction but other reactions are also possible. The grey color of the product suggests that some reduction to metallic aluminum occurs also. The insoluble nature of the solid could be consistent with an α -hydride elimination reaction. The thermal decomposition¹⁴ of Al(CH₃)₃ at 298°C yields methane and small quantities of ethane, ethylene and hydrogen by apparent α -hydride reactions. The hydrolysis of the grey solid from thermal decomposition of 1 mmol KAl(CH₃)₃H produces 2.58 mmol of a gas which is principally CH₄ but also contains small amounts of H₂. The predicted hydrolysis products, if only KAl(CH₃)₂ had been present, would be 2 mol CH₄ plus 1 mol H₂. The thermal decomposition of 1 mmol KAl(CH₃)₃H at higher temperatures, 200-230°C, for 12 hr. produced 1.18 mmol of a gas mixture consisting largely of CH₄ but also a small quantity of H₂. Thus, the thermal decomposition reactions of organo-aluminum(III) hydrides are complex and do not lead to the quantitative formation of an aluminum(I) species.

The possible reactions of Ga(CH₃)₃ with alkali-metal hydrides have also been investigated. Trimethylgallium reacts with KH in dimethoxy-

ethane or benzene at 25°C to form $\text{KGa}(\text{CH}_3)_3\text{H}$, a colorless solid (MP 150-152°C). The characteristic Ga-H stretching frequency is a sharp band at 2292 cm^{-1} . Since spectroscopic data confirmed the existence of a gallium hydride and no CH_4 was generated in the preparative reaction, the proposed reductive elimination did not occur at 25°C. When a sample of $\text{KGa}(\text{CH}_3)_3\text{H}$ was heated at 110° for 72 hr, no CH_4 or other evidence of reaction was observed. However, more extensive heating at 160°C led to the formation of CH_4 . After 72 hr, 0.984 mmol CH_4 /mmol $\text{KGa}(\text{CH}_3)_3\text{H}$ was formed. Heating for another 6 days produced an additional 0.506 mmol CH_4 /mmol $\text{KGa}(\text{CH}_3)_3\text{H}$. The solid remaining in the pyrolysis tube had a grey-black color and a "silver" colored mirror of gallium metal coated the walls of the tube. The grey solid did not have any Ga-H bonds according to its infrared spectrum. All of these results suggest that a well-defined product is not formed by a simple reductive elimination reaction.

The reaction of NaH with $\text{Ga}(\text{CH}_3)_3$ was investigated in order to study the effects of the apparent reducing agent. Results which were different from those observed for KH were obtained. When 1 mmol of $\text{Ga}(\text{CH}_3)_3$ and NaH were combined in dimethoxyethane at 25°C, 0.385 mmol of CH_4 was formed in 4 days. After the solvent was removed, the resultant mixture was heated at 135°C for 72 hr. and an additional 0.216 mmol CH_4 was formed. Further heating produced more CH_4 ; 145°C for 72 hr gave 0.124 mmol CH_4 ; 160°C for 120 hr led to another 0.269 mmol CH_4 . In total, 0.994 mmol CH_4 was formed. The material remaining in the tube after pyrolysis consisted of a grey solid, a viscous liquid which was soluble in dimethoxyethane, and a "silver" colored mirror of gallium

metal. These results suggest extensive decomposition and partial reduction of the sample to gallium metal. It is interesting that KH and NaH give slightly different results. However, there is no evidence for the formation of a characterizable product by a simple, well-defined reductive elimination reaction.

The observed chemistry of $\text{In}(\text{CH}_3)_3$ with NaH and KH lead to the same conclusions as those proposed for $\text{Al}(\text{CH}_3)_3$ and $\text{Ga}(\text{CH}_3)_3$. When 1 mmol $\text{In}(\text{CH}_3)_3$ was combined with excess KH in benzene at 25°C, 0.159 mmol of CH_4 and a grey-black solid was formed after 12 hr. The reaction of $\text{In}(\text{CH}_3)_3$ with NaH in dimethoxyethane at 130°C for 12 hr produced 0.120 mmol CH_4 and a black insoluble solid. It is of interest to compare our results with those of Gavrilenko, Kolesov and Zakharin.¹⁵ They reported that $\text{In}(\text{CH}_3)_3$ reacted with NaH and KH to form $\text{NaIn}(\text{CH}_3)_3\text{H}$ and $\text{In}(\text{CH}_3)_3\text{H}$. Heating to 100°C led to the formation of $\text{NaIn}(\text{CH}_3)_4$. It is difficult to reconcile our results but all data suggest that a simple, stoichiometric, reductive elimination reaction is not the only reaction occurring.

All of the results from the investigations of the reactions of Group 3 organometallic compounds with alkali-metal hydrides suggest that reductive elimination reactions can occur. The facility of the reaction depends on the alkali-metal hydride, the organic substituent and the Group 3 element. However, low oxidation state compounds are not the only product. The high temperatures required to initiate the reductive elimination reaction for the aluminum compounds and the methyl derivatives lead to apparent decomposition reactions which occur prior to or after the reduction reaction.

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